

## PATENT ABSTRACTS OF JAPAN

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## (54) CONDUCTIVE ROLLER

## (57)Abstract:

PROBLEM TO BE SOLVED: To control a hardness of an elastic layer and provide a conductive roller whose brittleness of the elastic layer is improved.

SOLUTION: The conductive roller is provided with as essential components an isobutylene-based polymer having in the molecule at least one hydrosilanizable alkenyl group, a compound having at least two hydrosilyl groups in the molecule, a hydrosilanization catalyst and carbon black, and is obtained by hardening around a metallic shaft a composition containing two or more kinds of compounds having at least two hydrosilyl groups in the molecule.

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CLAIMS

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## [Claim(s)]

[Claim 1] The conductive roller which is made to harden the conductive constituent characterized by using following (A) - (D) as an indispensable component, and using two or more sorts of (B) components around a metal shaft, and is obtained.

(A) (Compound C) hydrosilylation (catalyst D) addition-of-conductivity matter which has two or more hydrosilyl radicals in the isobutylene system (polymer B) molecule which has in a molecule the alkenyl radical in which at least one hydrosilylation reaction is possible.

[Claim 2] (A) The conductive roller according to claim 1 with which the alkenyl radical in which a hydrosilylation reaction is possible exists in the end of this polymer among the polymer of a component.

[Claim 3] (A) The conductive roller according to claim 1 or 2 whose number average molecular weight of the isobutylene system polymer of a component is 1000-50000.

[Claim 4] (A) A conductive roller given in any 1 term of claims 1-3 whose total amount of the repeat unit originating in the isobutylene in a component is 50 % of the weight or more.

[Claim 5] (B) A conductive roller given in any 1 term of claims 1-4 whose compound of a component is the polyorgano hydrogen siloxane which has two or more hydrosilyl radicals in 1 molecule on the average.

[Claim 6] (B) A conductive roller given in any 1 term of claims 1-5 whose components are the compound which has five or more hydrosilyl radicals in 1 molecule on the average, and the compound which has 2-4 hydrosilyl radicals in 1 molecule on the average.

[Claim 7] (D) A conductive roller given in any 1 term of claims 1-6 whose addition-of-conductivity matter of a component is carbon black.

[Claim 8] The conductive roller characterized by being a conductive roller given in any 1 term of claims 1-7, and the degree of hardness (JIS A) by JIS6301A law being 30 degrees or less.

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## DETAILED DESCRIPTION

## [Detailed Description of the Invention]

[0001] [Field of the Invention] This invention relates to a conductive roller. It is related with the conductive roller which is made to harden the constituent which makes it come to contain two or more sorts of compounds which use as an indispensable component the compound which has two or more hydrosilyl radicals in the isobutylene system polymer which has in more detail the alkenyl radical in which at least one hydrosilylation reaction is possible in a molecule, and a molecule, a hydrosilylation catalyst, and the addition-of-conductivity matter, and have two or more hydrosilyl radicals in a molecule around a metal shaft, and is obtained.

[0002] [Description of the Prior Art] Patent public presentation is already carried out and the conductive roller with which an elastic layer is prepared using an alkenyl radical content isobutylene system polymer, the compound containing a hydrosilyl radical, a hydrosilylation catalyst, and the conductive constituent that consists of carbon black is well-known. In these conductivity rubber roller, the reduction in the degree of hardness of an elastic layer is important, and the technique for adjusting a degree of hardness was needed. Moreover, the rubber elastic layer of the conductive roller obtained from the conductive constituent which uses an isobutylene system polymer as a principal component was weak, and its reinforcement was not necessarily enough.

[0003] [Problem(s) to be Solved by the Invention] This invention offers the conductive roller with which it was made in view of this actual condition, and the degree of hardness of an elastic layer could be controlled, and the brittleness of an elastic layer has been improved.

[0004] [Means for Solving the Problem] The isobutylene system polymer which this invention person repeats research wholeheartedly that the above-mentioned technical problem should be solved, and has in a molecule the alkenyl radical in which at least one hydrosilylation reaction is possible. In the compound which has two or more hydrosilyl radicals in a molecule, a hydrosilylation catalyst, and the conductive constituent which consists of a component called the addition-of-conductivity matter, the compound which has two or more hydrosilyl radicals in a molecule by using two or more sorts together. It came to make a header and this invention for becoming improvable [the reduction in the degree of hardness of the rubber elastic layer of the conductive roller obtained, and the brittleness of an elastic layer].

[0005] That is, the conductive roller which is made to harden the conductive constituent which this invention consists of (Compound C) hydrosilylation (catalyst D) addition-of-conductivity matter which has two or more hydrosilyl radicals in the isobutylene system (polymer B) molecule which has the alkenyl radical in which at least one hydrosilylation reaction is possible in the (A) molecule, and contains two or more sorts of (B) components around a metal shaft, and is obtained is offered.

[0006] (A) the alkenyl radical with a desirable component in which a hydrosilylation reaction among the polymer are possible — the end of this polymer — existing — moreover — desirable — the number average molecular weight of the isobutylene system polymer in a component — 1000-50000 — it is — further — moreover, the total amount of the repeat unit originating in the isobutylene in a component is 50 % of the weight or more preferably.

[0007] (B) Preferably, a component is a polyorgano hydrogen siloxane which has two or more hydrosilyl radicals in 1 molecule on the average, and contains at least one sort of a compound which averages with at least one sort of a compound which has five or more hydrosilyl radicals in 1 molecule on the average preferably, and has 2-4 hydrosilyl radicals in 1 molecule.

[0008] Moreover, the addition-of-conductivity matter of the (D) component has desirable carbon black.

[0009] Furthermore, as for the conductive roller which is made to harden the above-mentioned conductive

constituent around a metal shaft, and is obtained again, it is desirable that the degree of hardness (JIS A) by JIS6301A law is 30 degrees or less.

[0010]

[Embodiment of the Invention] The (A) component used for this invention is an isobutylene system polymer which has in a molecule the alkenyl radical in which at least one hydrosilylation reaction is possible. An isobutylene system polymer means what the monomeric unit which makes the frame of a polymer becomes mainly from an isobutylene unit here. In this case, the monomeric unit in which all the monomers may be formed in from the isobutylene unit, and it has an isobutylene and copolymeric — an isobutylene system polymer — you may contain in 20% or less of range preferably especially 30% or less preferably [it is desirable and ] in under 50% (% of the weight and the following — the same) pan. However, in these polymers frame, especially the thing for which the repeat unit which constitutes the principal chain which does not contain carbon-carbon unsaturated bonds other than a ring substantially excluding this alkenyl radical from moisture resistance, weatherability, and a heat-resistant viewpoint consists of saturated hydrocarbon is desirable. moreover, a unit monomer by which double association after a polymerization like a polyene compound like butadiene, isoprene, 1,3-tetra-deca diene, 1,9-deca diene, 1, and 5-hexadiene remains in the isobutylene system polymer used as a (A) component in this invention in the range in which the purpose of this invention is attained — small quantity — you may make it contain in 10% or less of range preferably

[0011] As an example of a copolymerization component of making the principal chain frame of such an isobutylene system polymer For example, 1-butene, 2-butene, a 2-methyl-1-butene, a 3-methyl-1-butene, A pentene, 4-methyl-1-pentene, a hexene, a vinyl cyclohexane, The methyl vinyl ether, ethyl vinyl ether, isobutyl vinyl ether, Styrene, alpha methyl styrene, dimethyl styrene, p-t-butoxy styrene, p-hexenylstyrene, p-ant ROKISHI styrene, p-hydroxystyrene, beta-pinene, an indene, vinyl dimethylmethoxysilane, a vinyl trimethyl silane, Divinyl dimethoxysilane, divinyl dimethylsilane, 1,3-divinyl - 1, 1, 3, and 3-tetramethyl disiloxane, TORIBI nil methylsilane, a tetra vinyl silane, allyl compound dimethyl methoxysilane, An allyl compound trimethyl silane, dian: dimethoxysilane, diaryl dimethylsilane, gamma-methacryloyl oxypropyl trimethoxysilane, gamma-methacryloyl oxypropyl methyl dimethoxysilane, etc. are mentioned.

[0012] Moreover, it will not be limited especially if the alkenyl radical in which a hydrosilylation reaction is possible is a radical including carbon-carbon duplex association which has activity to a hydrosilylation reaction. As an alkenyl radical, ring type unsaturated hydrocarbon radicals, such as aliphatic series partial saturation hydrocarbon groups, such as a vinyl group, an allyl group, a methylvinyl radical, a propenyl radical, a butenyl group, a pentenyl radical, and a hexenyl radical, a cyclo propenyl radical, a cyclo butenyl group, a cyclo pentenyl radical, and a cyclohexenyl group, a methacrylic radical, etc. are mentioned. As for the (A) component in this invention, it is desirable to introduce into the polymer and the alkenyl radical in which the above-mentioned hydrosilylation reaction is possible. Thus, when an alkenyl radical is in a polymer end, it is desirable from points, like the rubber-like hardened material of high intensity becomes the effective network chain density of the hardened material finally formed increases, and is easy to be obtained.

[0013] As for the number average molecular weight (the GPC method, polystyrene conversion) of said isobutylene system polymer, it is desirable that it is 1000 to about 50000, and what has especially the liquefied object which is 2000 to about 30000, and a fluidity is desirable from a viewpoint of the ease of dealing with it et

[0014] The approach of introducing an alkenyl radical into the isobutylene system polymer which has a covalent bond nature Cl radical as the manufacture approach of the (A) component of this invention, for example is mentioned. Although there is especially no limit in the approach of introducing an alkenyl radical into the isobutylene system polymer which has a covalent-bond nature Cl radical For example, the method of performing a Friedel Crafts reaction between alkenyl phenyl ether and Cl radical of a polymer. After performing the Friedel Crafts reaction of the approach of carrying out the substitution reaction of an allyl compound trimethyl silane etc. and the Cl radical of a polymer under Lewis acid existence, and various phenols and Cl radical of a polymer and introducing a hydroxyl group, the method of performing the further aforementioned alkenyl radical installatic approach etc. is mentioned.

[0015] Next, how to introduce an alkenyl radical during the polymerization of a polymer is explained.

[0016] As an approach of introducing an alkenyl radical during the polymerization of a polymer, an allyl compound trimethyl silane is added to the system of reaction to which the cationic polymerization of the cationic polymerization nature monomer containing an isobutylene is carried out under existence of an initiator, a chain transfer agent, and a catalyst, and there is a method of manufacturing the isobutylene system polymer which has an allyl compound end, for example.

[0017] Here, the compound which has the halogen atom combined with the compound or tertiary carbon atom which has the carbon atom combined with a halogen atom and ring carbon as for example, an initiator-cum-a

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JP,2002-075053,A [DETAILED DESCRIPTION]

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chain transfer agent can be used. Lewis acid can be used as a catalyst.

[0018] Similarly, the approach of adding the nonconjugated diene like 1 and 9-deca diene or the alkenyloxy styrene like p-hexenylstyrene to the polymerization system of reaction is also possible.

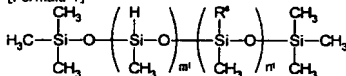
[0019] Here, although the compounds (for example, BCl<sub>3</sub>, Et<sub>2</sub>AlCl, EtAlCl<sub>2</sub>, AlCl<sub>3</sub>, SnCl<sub>4</sub>, TiCl<sub>4</sub>, VCl<sub>5</sub>, FeCl<sub>3</sub>, BF<sub>3</sub>, etc.) expressed with general formula MX<sub>n</sub> (M is a metal atom and X<sup>\*</sup> is a halogen atom) are mentioned as Lewis acid which may be used as a cationic polymerization catalyst, it is not limited to these. BCl<sub>3</sub>, SnCl<sub>4</sub>, BF<sub>3</sub>, etc. are TiCl<sub>4</sub>(s)4 desirable still more preferably among these Lewis acid. To the number of mols of an initiator-cum-a chain transfer agent, 0.1 to 10 times are desirable still more desirable, and it is 2 to 5 times the amount of the Lewis acid used of this.

[0020] As a compound which has the hydrosilyl radical which is the (B) component of this invention, the compound of the arbitration which contains two or more hydrosilyl radicals in intramolecular is usable. Here, a "hydrosilyl radical" is a silyl radical expressed with general formula: HnR(3-n) Si<sup>\*</sup> (however, R is the radical of the monovalence of arbitration), and the radical which has the hydrogen atom combined with the silicon atom concerned is said. In this detail in the letter, it is alike and sets, and for convenience, when two hydrogen atoms (H) have combined with the same silicon atom (Si), it calculates with two hydrosilyl radicals.

[0021] (B) A polyorgano hydrogen siloxane is mentioned as a desirable example of the compound of a component. A polyorgano hydrogen siloxane means the siloxane compound which has a hydrocarbon group or a hydrogen atom on a silicon atom. A siloxane compound may be the shape for example, of a chain, or annular, and is shown by the following concrete for example, formulas.

[0022]

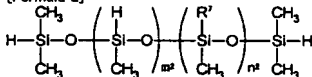
[Formula 1]



(It is 2 <=m1, is 0 <=n1, and is 2 <=m1+n1 <=50, and R<sup>\*</sup> is a hydrocarbon except the alkenyl radical of carbon numbers 2-20, and an alkenyl group, and R<sup>\*</sup> may be permuted by one or more phenyl groups if needed.)

[0023]

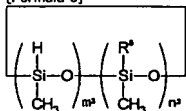
[Formula 2]



(It is 0 <=m2, is 0 <=n2, and is 0 <=m2+n2 <=50, and R<sup>\*</sup> is a hydrocarbon except the alkenyl radical of carbon numbers 2-20, and an alkenyl group, and R<sup>\*</sup> may be permuted by one or more phenyl groups if needed.)

[0024]

[Formula 3]



(It is 2 <=m3 <=19, is 0 <=n3 <=18, and is 3 <=m3+n3 <=20, R<sup>\*</sup> is the hydrocarbon of carbon numbers 2-20, and R<sup>\*</sup> may be permuted by one or more phenyl groups if needed. Or it has the shape of a chain shown, annular things, and these two or more units, a siloxane compound may have two or more siloxane units, and is shown by the following concrete for example, formulas.

[0025]

[Formula 4]

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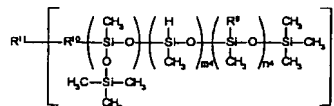
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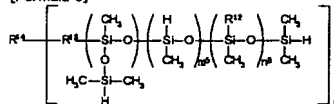
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(Here, it is 1 <=m4, is 0 <=n4, and is 1 <=m4+n4 <=50, and R<sup>\*</sup> is the hydrocarbon of carbon numbers 2-20, and R<sup>\*</sup> may be permuted by one or more phenyl groups if needed.) It is 2 <=l, and R<sup>10</sup> is a divalent organic radical, or R<sup>10</sup> does not need to exist. R<sup>11</sup> is the organic radical of 2 - tetravalence.

[0026]

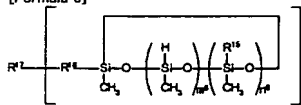
[Formula 5]



(It is 0 <=m5, is 0 <=n5, and is 0 <=m5+n5 <=50, and R<sup>12</sup> is the hydrocarbon of carbon numbers 2-20, and R<sup>12</sup> may be permuted by one or more phenyl groups if needed.) It is 2 <=l2, and R<sup>13</sup> is a divalent organic radical, or R<sup>13</sup> does not need to exist. R<sup>14</sup> is the organic radical of 2 - tetravalence.

[0027]

[Formula 6]



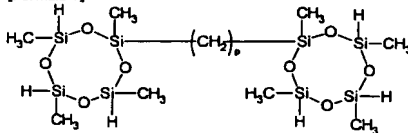
(It is 1 <=m6, is 0 <=n6, and is 3 <=m6+n6 <=50, and R<sup>15</sup> is the hydrocarbon of carbon numbers 2-20, and R<sup>15</sup> may be permuted by one or more phenyl groups if needed.) It is 2 <=l3, and R<sup>16</sup> is a divalent organic radical, or R<sup>16</sup> does not need to exist. R<sup>17</sup> is the organic radical of 2 - tetravalence.

(B) As for a component, what has good compatibility with the (A) component, the (C) component, and the (D) component or the distributed stability in a system is desirable. If compatibility with each above-mentioned component of the (B) component is low when especially the viscosity of the whole system is low, phase separation will tend to happen and it will be easy to cause poor hardening. For this reason, in order to prevent phase separation, a filler with small particle size, such as an impalpable powder silica, may be blended as a distributed assistant.

[0028] The following concrete for example, compounds are mentioned as a (B) component with comparative good compatibility or distributed stability with a component, the (C) component, or the (D) component.

[0029]

[Formula 7]



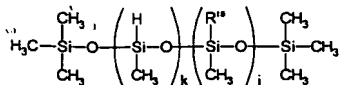
(Here, p is the integer of 6-12.)

[0030]

[Formula 8]

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(It is  $2 < k < 10$ , and is  $0 < j < 5$ , and R<sup>18</sup> is a with a carbon numbers of eight or more hydrocarbon group.)  
 (B) As for the amount of the component used, it is desirable to use it to the total number of mols of the alkenyl radical of the (A) component, so that the total number of mols of the silicon atomic union hydrogen atom of the (B) component may become 0.8–5.0Eq. To the alkenyl radical total amount of the above-mentioned (A) component, when there are too few silicon atomic union hydrogen atoms of the (B) component, bridge formation tends to become inadequate. Moreover, in many [ too ], a silicon atomic union hydrogen atom tends to remain after hardening, and physical properties tend to change with the effects of the hydrogen atom a lot. It is desirable to make the amount of the (B) component into 1.0–2.0Eq to control especially this effect.

[0031] Here, the substituent of the above-mentioned general formula is explained.  
 [0032] A hydrocarbon expresses an alkyl group, an alkenyl radical, an alkynyl group, an aryl group, an aralkyl radical, and a cycloalkyl radical preferably, when there is no notice especially.  
 [0033] as an alkyl group — desirable — carbon numbers 1–30 — more — desirable — carbon numbers 1–20 — the methyl of carbon numbers 1–10, ethyl, propyl, etc. are mentioned still more preferably.  
 [0034] as an alkenyl radical — desirable — carbon numbers 2–30 — more — desirable — carbon numbers 2–20 — the vinyl of carbon numbers 2–10, an allyl compound, etc. are mentioned still more preferably.  
 [0035] as an alkynyl group — desirable — carbon numbers 2–30 — more — desirable — carbon numbers 2–20 — the ethynyl of carbon numbers 2–10, propargyl, etc. are mentioned still more preferably.  
 [0036] as an aryl group — desirable — carbon numbers 6–30 — more — desirable — carbon numbers 6–20 — the phenyl of carbon numbers 6–10, tolyl, naphthyl, etc. are mentioned still more preferably.  
 [0037] as an aralkyl radical — desirable — carbon numbers 7–30 — more — desirable — carbon numbers 7–20 — the benzyl of carbon numbers 7–10, phenethyl, etc. are mentioned still more preferably.  
 [0038] as a cycloalkyl radical — desirable — carbon numbers 3–10 — the cyclopentyl of carbon numbers 3–8, cyclohexyl, etc. are mentioned more preferably.  
 [0039] as an alkoxy group — desirable — carbon numbers 1–30 — more — desirable — carbon numbers 1–20 — the methoxy of carbon numbers 1–10, ethoxy, propoxy, etc. are mentioned still more preferably.  
 [0040] as a divalent organic radical — O and carbon numbers 1–10 — the methylene of carbon numbers 1–3, ethylene, etc. are mentioned preferably.  
 [0041] In addition to the above-mentioned divalent organic radical, as an organic radical of 2 — tetravalence, N and C are mentioned, for example.  
 [0042] It is using two or more sorts together, and the (B) component in this invention can control the degree of hardness of a conductive roller, further, it can improve brittleness and offer of a good conductive roller of it is attained. Here, as for the (B) component, it is desirable to use together one or more sorts of a compound which average with one or more sorts of a compound which have five or more hydrosilyl radicals in 1 molecule on the average, and have 2–4 hydrosilyl radicals in 1 molecule. (B) In a component, although crosslinking density becomes small and can improve the brittleness of the hardened material obtained, and an elongation property so that the number of the hydrosilyl radicals in a molecule decreases (it has 2–4 hydrosilyl radicals preferably), increase of tack nature is invited to coincidence and there is a possibility of becoming a problem. In order not to cause increase of tack nature, as for at least one sort of the hydrosilyl radical content compound to be used, it is desirable to have five or more hydrosilyl radicals in intramolecular.  
 [0043] (C) As a hydrosilylation catalyst of a component, there is especially no limit and it can use the hydrosilylation catalyst of arbitration. the catalyst: platinum–vinyl siloxane complex which made support, such as chloroplatinic acid, a simple substance of platinum, an alumina, a silica, or carbon black, support solid-state platinum when illustrating concretely — [— for example Pt<sub>2</sub>(ViMe<sub>2</sub>SiOSiMe<sub>2</sub>Vi) n and a Pt[(MeViSiO)<sub>4</sub>] m]; platinum–phosphine complex — [— for example a Pt(PPh<sub>3</sub>)<sub>4</sub> and Pt(PBu<sub>3</sub>)<sub>4</sub>]; platinum–phosphite complex — [— for example Pt[P(OPh)<sub>3</sub>] 4 and Pt[P(OBu)<sub>3</sub>] 4 (the inside of a formula, and Me — a methyl group —) in Bu, butyl and Vi express a vinyl group, Ph expresses a phenyl group, and n and m express an integer —] The platinum alcoholate catalyst indicated in the platinum–hydrocarbon complex indicated in Pt(acac)<sub>2</sub>, Ashby's and others United States patent 3159601st, and a No. 3159662 specification and Lamoreaux's and others U.S. Pat. No. 3220972 specification is also mentioned.  
 [0044] Moreover, as an example of catalysts other than a platinum compound, RhCl (PPh<sub>3</sub>)<sub>3</sub>, RhCl<sub>3</sub>, Rh/aluminum 2O<sub>3</sub>, RuCl<sub>3</sub>, IrCl<sub>3</sub>, FeCl<sub>3</sub> and AlCl<sub>3</sub>, PdCl<sub>2</sub>·2H<sub>2</sub>O, NiCl<sub>2</sub>, TiCl<sub>4</sub>, etc. are mentioned. These catalysts

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may be used independently, and even if it uses two or more sorts together, they are not cared about.  
 Chloroplatinic acid, a platinum–olefin complex, a platinum–vinyl siloxane complex, and Pt(acac)<sub>2</sub> grade are desirable from the point of catalytic activity.  
 [0045] (C) Especially the amount of the catalyst of a component has the desirable range of 10–8 to ten to 1 mol to one mol of alkenyl radicals in the (A) component, although not restricted. It is the range of 10–6 to ten to 2 mol more preferably. Moreover, it is better many [ since a hydrosilylation catalyst is generally expensive, and it is corrosive, and hydrogen gas is generated in large quantities and a hardened material may foam / too ].  
 [0046] (D) A component is a component for giving conductivity to the conductive roller of this invention. (D) As addition-of-conductivity matter of a component, carbon black, a metallic oxide, metal impalpable powder, etc. are mentioned. Moreover, the organic compound and polymer which have quaternary ammonium salt, a carboxylic-acid radical, a sulfonic group, a sulfate radical, a phosphoric ester radical, etc. can also serve as addition-of-conductivity matter. Furthermore, the compound classified into an antistatic agent depending on the cases, such as a compound, a polymer, etc. which have the conductive unit represented with ether ester imide, an ether imide polymer, an ethyleneoxide–epihalohydrin copolymer, methoxy polyethylene–glycol acrylate, etc., can also serve as addition-of-conductivity matter.  
 [0047] Here, also when checking a hydrosilylation reaction depending on the class or addition of the addition-of-conductivity matter to be used, for a certain reason, the effect to the hydrosilylation reaction of the addition-of-conductivity matter must be taken into consideration. In addition, the (D) component may be used independently and may use two or more sorts together.  
 [0048] As an example of the above-mentioned carbon black, furnace black, acetylene black, lamp black, channel black, thermal black, oil black, etc. are mentioned. There is no limit in the class of these carbon black, particle size, etc., and it is suitably chosen as them according to the engine performance of a request of a conductive roller etc.  
 [0049] (D) The addition of a component can be chosen as arbitration according to the conductivity needed for a conductive roller. Preferably, it is the 0.1 – 200 weight section to the polymer 100 weight section of the (A) component, and the 1 – 100 weight section is more desirable. If there are too few additions, it will become easy to come to the conductivity of the conductive ingredient obtained out of variation. On the other hand, if an addition increases too much, the fluidity of a constituent will tend to fall and workability will tend to fall.  
 [0050] Moreover, on the conductive roller of this invention, a softener and a plasticizer may be added in order to adjust viscosity and a degree of hardness. Below the 150 weight sections of the amount of a softener and the plasticizer used are desirable to the (A) component 100 weight section. Problems, such as bleeding, may be produced if it becomes an addition beyond it.  
 [0051] Moreover, a storage stability amelioration agent can be used for the constituent which forms the elastic layer of the conductive roller of this invention in order to improve storage stability. It is not limited especially that to be the usual stabilizer known as a preservation stabilizer of the (B) component of this invention as this storage stability amelioration agent, and what is necessary is just what attains the desired end. Specifically, the compound containing an aliphatic series unsaturated bond, an organic phosphorus compound, an organosulfur compound, a nitrogen content compound, a tin system compound, organic peroxide, etc. can be used suitably. Still more specifically 2-benzothiazolyl sulfide, benzothiazole, A thiazole, dimethyl acetylene di carboxylate, diethyl acetylene di carboxylate, Butylhydroxytoluene, butylhydroxyanisole, vitamin E, 2-(4-mol FOJL nil dithio) benzothiazole, 3-methyl-1-butene-3-ol, An acetylene nature partial saturation radical content ORGANO siloxane, an ethylene nature partial saturation radical content ORGANO siloxane, Although acetylene alcohol, 3-methyl-1-butyl-3-ol, diallyl fumarate, diallyl maleate, diethyl fumarate, diethyl maleate, dimethylmaleate, 2-pentene nitril, 2, and 3-dichloropropene etc. is mentioned It is not necessarily limited to these.  
 [0052] Moreover, to the constituent which forms the elastic layer of the conductive roller of this invention, various functional grant agents, such as various bulking agents and an adhesive grant agent, an antioxidant, an ultraviolet ray absorbent, a pigment, a surfactant, a solvent, and a silicon compound may be added suitably. Various silane coupling agents are illustrated as said adhesive grant agent. The effect of especially the silane coupling agent that has functional groups, such as an epoxy group, a methacryloyl radical, and a vinyl group, affect hardenability is also small, and it is easy to use effectiveness also for an adhesive manifestation greatly. However, as a silane coupling agent which can be used, it is not limited to these.  
 [0053] Especially the manufacture approach of the conductive roller in this invention is not limited, but the shaping approach of various well-known rollers can be conventionally used for it. For example, a conductive constituent is fabricated by the various fabricating methods, such as extrusion molding, press forming, injection molding, reaction injection molding (RIM), liquefied injection molding (LIM), and cast molding, the metal mold which installed metal shafts, such as a product made from SUS, in the core is made to carry out heat hardening by suitable temperature and time amount, and a conductive elastic layer is fabricated around a shaft to it. Here,

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# JP,2002-075053A [DETAILED DESCRIPTION]

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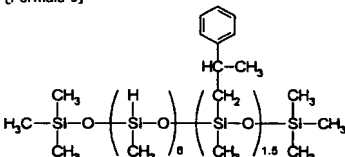
# JP,2002-075053A [DETAILED DESCRIPTION]

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as the manufacture approach of the conductive roller in this invention, since the conductive constituent for forming an elastic layer is liquefied, liquefied injection molding is desirable in respect of productivity and workability. In this case, after carrying out semi-hardening of the conductive constituent, it may establish and carry out full hardening of the process which carries out posture separately. Furthermore, an unit or two or more layers may be prepared in the outside of said conductive elastic layer if needed. For example, from said conductive elastic layer, the resin for surface layer formation can be applied to predetermined thickness a spray coating cloth and by roll-coat-applying or DIP applying, it can be made to be able to dry and harden at predetermined temperature, and a surface layer can be prepared.  
 [0054] In this invention, since a constituent hardens by the addition reaction of the Si–H radical to the alkenyl radical using a precious metal catalyst, a cure rate is very quick, and when performing Rhine production, it is convenient. Especially the temperature that carries out heat curing has desirable within the limits of 80 degrees C – 200 degrees C.  
 [0055] Moreover, it sets to this invention and a roller degree of hardness is JIS. It measures based on K6301A law. Specifically, it measures about the conductive roller by this invention, using A mold hardness tester as a measuring instrument. Here, the degree of hardness of the conductive roller in this invention is JIS because of mitigation of toner stress etc. 30 degrees or less and 25 more degrees or less have desirable A degree of hardness.  
 [0056]  
 [Example] Although this invention is further explained to a detail based on the following examples, this invention is not limited only to these examples.  
 [0057] (Example 1) As a (A) component, it is [ carbon black (Mitsubishi Chemical #3030B) 30g and ] MARK as an antioxidant to 400A(Kaneka make) 300g of allyl group end polyisobutylenes EP as 150g and a plasticizer PAO5006(product made from Idemitsu petrochemistry) (D) component. AO–50(product made from the Asahi electrofinition) 3g was mixed, and it kneaded 3 times with a roll. Subsequently, it is 4g (the hydrosilyl radical to contain is the 0.5-mol equivalent to the allyl group of the (A) component) about the compound B1 which shows structure below to this mixture as a (B) component.

[0058]

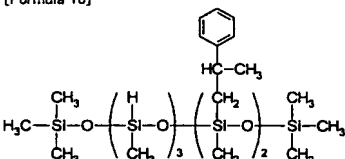
[Formula 9]



And 22g (hydrosilyl radical to contain is the 1.5-mol equivalent to allyl group of (A) component) mixing of compound B–2 which shows structure below was carried out.

[0059]

[Formula 10]



Furthermore, the bis(1, 3-divinyl – 1, 1, 3, and 3-tetramethyl disiloxane) platinum complex compound catalyst (17.9x10–5 mmol/mol, xylene solution) was \*\*\* (ed) as a (C) component, the 1-ethynyl-1-cyclohexanol was \*\*\* (ed) 0.9g as 170microl, and a storage stability amelioration agent, and homogeneity mixing was carried out. About this constituent, after warming at 50 degrees C, degassing was carried out with vacuum mixing degassing equipment for 5 hours. This conductive constituent was poured into the roller molding die by injection pressure

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1MPa, and the conductive roller which prepared the conductive rubber layer with 3mm [ in thickness ] and a die length of 230mm in the surroundings of the shaft with a diameter of 8mm made from SUS was created. About the obtained roller, it is JIS at the conditions of the temperature of 23 degrees C, and 65% of humidity. The rolle degree of hardness (JIS A) by K6301A law was measured. The result was shown in Table 1.  
 [0060] (Example 2) After filling up the metal mold frame of the aluminum which covered with the Teflon sheet with the conductive constituent obtained by the formula of example 1 publication, for 150 degrees C and 30 minutes, press forming was carried out on condition that heating, and the sheet-like hardened material for evaluation of 2mm thickness was obtained. the obtained sheet-like hardened material — the shuttering for 2 (1/3) number types — piercing — the constant temperature of the temperature of 23 degrees C, and 65%\*5% of: humidity — tension rate 200 mm/min and the distance between chucks of 20mm estimated breaking strength and the maximum elongation under the constant humidity condition using Shimazu autograph AG–2000A. A resu is shown in Table 1.  
 [0061] (Example 3) About the formula of example 1 publication, as a (B) component, 15g (hydrosilyl radical to contain is the 1.0-mol equivalent to allyl group of (A) component) combination of 8g (the hydrosilyl radical to contain is the 1.0-mol equivalent to the allyl group of the (A) component) and compound B–2 was carried out to the compound B1, and the rest evaluated by creating a conductive roller similarly. A recipe and a result are shown in Table 1.  
 [0062] (Example 4) Using the conductive constituent used in the example 3, by the approach of example 2 publication, the sheet-like hardened material was created and same evaluation was performed. A result is shown in Table 1.  
 [0063] (Example 1 of a comparison) About the formula of example 1 publication, as a (B) component, 16g (hydrosilyl radical to contain is the 2.0-mol equivalent to allyl group of (A) component) combination only of the compound B1 was carried out, and the rest evaluated by creating a conductive roller similarly. A recipe and a result are shown in Table 1.  
 [0064] (Example 2 of a comparison) Using the conductive constituent used in the example 1 of a comparison, by the approach of example 2 publication, the sheet-like hardened material was created and same evaluation was performed. A result is shown in Table 1.

[0065]

[Table 1]

		実施例1, 2	実施例3, 4	比較例1, 2
(A) 成分	EP400A	g	300	300
(B) 成分	化合物B1	g	4	8
	化合物B2	g	22	15
(C) 成分	Pt: 2,2,6,6-テトラメチルシクロヘキサン-1,4-ジオール	μL	170	170
(D) 成分	PAO5006	g	30	30
可塑剤	PAO5006	g	150	150
貯蔵安定性改良剤	1-エチン-1-シクロヘキサン	g	0.9	0.9
硬化防止剤	MARK AO-50	g	3	3
評価	ロ-硬度		11	13
	破断強度	kg/cm <sup>2</sup>	6.9	7.8
	最大伸び	%	330	310

[0066]

[Effect of the Invention] It became possible to be able to lower the degree of hardness of the elastic layer of a conductive roller, and to improve brittleness by this invention, by using into a molecule two or more sorts of compounds which have two or more hydrosilyl radicals.

[Translation done.]

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## TECHNICAL FIELD

[Field of the Invention] This invention relates to a conductive roller. It is related with the conductive roller which is made to harden the constituent which makes it come to contain two or more sorts of compounds which use as an indispensable component the compound which has two or more hydrosilyl radicals in the isobutylene system polymer which has in more detail the alkenyl radical in which at least one hydrosilylation reaction is possible in a molecule, and a molecule, a hydrosilylation catalyst, and the addition-of-conductivity matter, and have two or more hydrosilyl radicals in a molecule around a metal shaft, and is obtained.

[Translation done.]

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JP,2002-075053,A [EFFECT OF THE INVENTION]

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## EFFECT OF THE INVENTION

[Effect of the Invention] It became possible to be able to lower the degree of hardness of the elastic layer of a conductive roller, and to improve brittleness by this invention, by using into a molecule two or more sorts of compounds which have two or more hydrosilyl radicals.

[Translation done.]

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## PRIOR ART

[Description of the Prior Art] Patent public presentation is already carried out and the conductive roller with which an elastic layer is prepared using an alkenyl radical content isobutylene system polymer, the compound containing a hydrosilyl radical, a hydrosilylation catalyst, and the conductive constituent that consists of carbon black is well-known. In these conductivity rubber roller, the reduction in the degree of hardness of an elastic layer is important, and the technique for adjusting a degree of hardness was needed. Moreover, the rubber elastic layer of the conductive roller obtained from the conductive constituent which uses an isobutylene system polymer as a principal component was weak, and its reinforcement was not necessarily enough.

[Translation done.]

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JP,2002-075053,A [TECHNICAL PROBLEM]

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## TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] This invention offers the conductive roller with which it was made in view of this actual condition, and the degree of hardness of an elastic layer could be controlled, and the brittleness of an elastic layer has been improved.

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## MEANS

[Means for Solving the Problem] The isobutylene system polymer which this invention person repeats research wholeheartedly that the above-mentioned technical problem should be solved, and has in a molecule the alkenyl radical in which at least one hydrosilylation reaction is possible. In the compound which has two or more hydrosilyl radicals in a molecule, a hydrosilylation catalyst, and the conductive constituent which consists of a component called the addition-of-conductivity matter, the compound which has two or more hydrosilyl radicals in a molecule by using two or more sorts together it came to make a header and this invention for becoming improvable [the reduction in the degree of hardness of the rubber elastic layer of the conductive roller obtained, and the brittleness of an elastic layer].

[0005] That is, the conductive roller which is made to harden the conductive constituent which this invention consists of (Compound C) hydrosilylation (catalyst D) addition-of-conductivity matter which has two or more hydrosilyl radicals in the isobutylene system (polymer B) molecule which has the alkenyl radical in which at least one hydrosilylation reaction is possible in the (A) molecule, and contains two or more sorts of (B) components around a metal shaft, and is obtained is offered.

[0006] (A) the alkenyl radical with a desirable component in which a hydrosilylation reaction among the polymer are possible — the end of this polymer — existing — moreover — desirable — the number average molecular weight of the isobutylene system polymer in a component — 1000-50000 — it is — further — moreover, the total amount of the repeat unit originating in the isobutylene in a component is 50 % of the weight or more preferably.

[0007] (B) Preferably, a component is a polyorgano hydrogen siloxane which has two or more hydrosilyl radicals in 1 molecule on the average, and contains at least one sort of a compound which averages with at least one sort of a compound which has five or more hydrosilyl radicals in 1 molecule on the average preferably, and has 2-4 hydrosilyl radicals in 1 molecule.

[0008] Moreover, the addition-of-conductivity matter of the (D) component has desirable carbon black.

[0009] Furthermore, as for the conductive roller which is made to harden the above-mentioned conductive constituent around a metal shaft, and is obtained again, it is desirable that the degree of hardness (JIS A) by JIS6301A law is 30 degrees or less.

[0010] [Embodiment of the Invention] The (A) component used for this invention is an isobutylene system polymer which has in a molecule the alkenyl radical in which at least one hydrosilylation reaction is possible. An isobutylene system polymer means what the monomeric unit which makes the frame of a polymer becomes mainly from an isobutylene unit here. In this case, the monomeric unit in which all the monomers may be formed in from the isobutylene unit, and it has an isobutylene and copolymeric — an isobutylene system polymer — you may contain in 20% or less of range preferably especially 30% or less preferably [it is desirable and ] in under 50% (% of the weight and the following — the same) pan. However, in these polymers frame, especially the thing for which the repeat unit which constitutes the principal chain which does not contain carbon-carbon unsaturated bonds other than a ring substantially excluding this alkenyl radical from moisture resistance, weatherability, and a heat-resistant viewpoint consists of saturated hydrocarbon is desirable. moreover, a unit monomer by which double association after a polymerization like a polyene compound like butadiene, isoprene, 1, 13-tetra-deca diene, 1, 9-deca diene, 1, and 5-hexadiene remains in the isobutylene system polymer used as a (A) component in this invention in the range in which the purpose of this invention is attained — small quantity — you may make it contain in 10% or less of range preferably

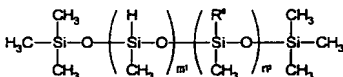
[0011] As an example of a copolymerization component of making the principal chain frame of such an isobutylene system polymer For example, 1-butene, 2-butene, a 2-methyl-1-butene, a 3-methyl-1-butene, A pentene, 4-methyl-1-pentene, a hexene, a vinyl cyclohexane, The methyl vinyl ether, ethyl vinyl ether, isobutyl vinyl ether, Styrene, alpha methyl styrene, dimethyl styrene, p-t-butoxy styrene, p-hexenyloxy styrene, p-ant

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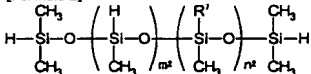
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(It is  $2 \leq m \leq 1$ , is  $0 \leq n \leq 1$ , and is  $2 \leq m+n \leq 50$ , and R6 is a hydrocarbon except the alkenyl radical of carbon numbers 2-20, and an alkynyl group, and R6 may be permuted by one or more phenyl groups if needed.)

[0023]

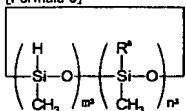
[Formula 2]



(It is  $0 \leq m \leq 2$ , is  $0 \leq n \leq 2$ , and is  $0 \leq m+n \leq 50$ , and R7 is a hydrocarbon except the alkenyl radical of carbon numbers 2-20, and an alkynyl group, and R7 may be permuted by one or more phenyl groups if needed.)

[0024]

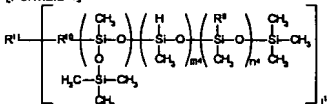
[Formula 3]



(It is  $2 \leq m \leq 19$ , is  $0 \leq n \leq 18$ , and is  $3 \leq m+n \leq 20$ , R8) it is the hydrocarbon of carbon numbers 2-20, and R8 may be permuted by one or more phenyl groups if needed. Or it has the shape of a chain shown, annular things, and these two or more units, a siloxane compound may have two or more siloxane units, and is shown by the following concrete for example, formulas.

[0025]

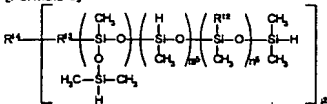
[Formula 4]



(Here, it is  $1 \leq m \leq 4$ , is  $0 \leq n \leq 4$ , and is  $1 \leq m+n \leq 50$ , and R9 is the hydrocarbon of carbon numbers 2-20, and R9 may be permuted by one or more phenyl groups if needed.) It is  $2 \leq k \leq 1$ , and R10 is a divalent organic radical, or R10 does not need to exist. R11 is the organic radical of 2 - tetravalence.

[0026]

[Formula 5]



(It is  $0 \leq m \leq 5$ , is  $0 \leq n \leq 5$ , and is  $0 \leq m+n \leq 50$ , and R12 is the hydrocarbon of carbon numbers 2-20, and R12 may be permuted by one or more phenyl groups if needed.) It is  $2 \leq k \leq 2$ , and R13 is a divalent organic radical, or R13 does not need to exist. R14 is the organic radical of 2 - tetravalence.

[0027]

[Formula 6]



ROKISHI styrene, p-hydroxystyrene, beta-pinene, an indene, vinyldimethylmethoxysilane, a vinyl trimethyl silane Divinyl dimethoxysilane, divinyl dimethylsilane, 1, 3-divinyl - 1, 1, 3, and 3-tetramethyl siloxane, TORIBI nil methylsilane, a tetravinyl silane, allyl compound dimethyl methoxysilane, An allyl compound trimethyl silane, diar: dimethoxysilane, diaryl dimethylsilane, gamma-methacryloyl oxypropyl trimethoxysilane, gamma-methacryloyl oxypropyl methyl dimethoxysilane, etc. are mentioned.

[0012] Moreover, it will not be limited especially if the alkenyl radical in which a hydrosilylation reaction is possible is a radical including carbon-carbon duplex association which has activity to a hydrosilylation reaction. As an alkenyl radical, ring type unsaturated hydrocarbon radicals, such as aliphatic series partial saturation hydrocarbon groups, such as a vinyl group, an allyl group, a methylvinyl radical, a propenyl radical, a butenyl group, a pentenyl radical, and a hexenyl radical, a cyclo propenyl radical, a cyclo butenyl group, a cyclo pentenyl radical, and a cyclohexenyl group, an methacrylic radical, etc. are mentioned. As for the (A) component in this invention, it is desirable to introduce into the polymer end the alkenyl radical in which the above-mentioned hydrosilylation reaction is possible. Thus, when an alkenyl radical is in a polymer end, it is desirable from points, like the rubber-like hardened material of high intensity becomes the effective network chain density of the hardened material finally formed increases, and is easy to be obtained.

[0013] As for the number average molecular weight (the GPC method, polystyrene conversion) of said isobutylene system polymer, it is desirable that it is 1000 to about 50000, and what has especially the liquefied object which is 2000 to about 30000, and a fluidity is desirable from a viewpoint of the ease of dealing with it et

[0014] The approach of introducing an alkenyl radical into the isobutylene system polymer which has a covalent bond nature Cl radical as the manufacture approach of the (A) component of this invention, for example is mentioned. Although there is especially no limit in the approach of introducing an alkenyl radical into the isobutylene system polymer which has a covalent-bond nature Cl radical For example, the method of performing a Friedel Crafts reaction between alkenyl phenyl ether and Cl radical of a polymer. After performing the Friede Crafts reaction of the approach of carrying out the substitution reaction of an allyl compound trimethyl silane etc. and the Cl radical of a polymer under Lewis acid existence, and various phenols and Cl radical of a polymer and introducing a hydroxyl group, the method of performing the further aforementioned alkenyl radical installatio approach etc. is mentioned.

[0015] Next, how to introduce an alkenyl radical during the polymerization of a polymer is explained.

[0016] As an approach of introducing an alkenyl radical during the polymerization of a polymer, an allyl compound trimethyl silane is added to the system of reaction to which the cationic polymerization of the cationic polymerization nature monomer containing an isobutylene is carried out under existence of an initiator, a chain transfer agent, and a catalyst, and there is a method of manufacturing the isobutylene system polymer which has an allyl compound end, for example.

[0017] Here, the compound which has the halogen atom combined with the compound or tertiary carbon atom which has the carbon atom combined with a halogen atom and ring carbon as for example, an initiator-cum-a chain transfer agent can be used. Lewis acid can be used as a catalyst.

[0018] Similarly, the approach of adding the nonconjugated diene like 1 and 9-deca diene or the alkenyloxy styrene like p-hexenyloxy styrene to the polymerization system of reaction is also possible.

[0019] Here, although the compounds (for example, BCl3, Et2AlCl, EtAlCl2, AlCl3, SnCl4, TiCl4, VCl5, FeCl3, BF3, etc.) expressed with general formula MX'n (M is a metal atom and X' is a halogen atom) are mentioned as Lewis acid which may be used as a cationic polymerization catalyst, it is not limited to these. BCl3, SnCl4, BF3, etc. are TiCl4(s)4 desirable still more preferably among these Lewis acid. To the number of mols of an initiator-cum-a chain transfer agent, 0.1 to 10 times are desirable still more desirable, and it is 2 to 5 times the amount of the Lewis acid used of this.

[0020] As a compound which has the hydrosilyl radical which is the (B) component of this invention, the compound of the arbitration which contains two or more hydrosilyl radicals in intramolecular is usable. Here, a "hydrosilyl radical" is a silyl radical expressed with general formula: HnR(3-n) Si• (however, R is the radical of t: monovalence of arbitration), and the radical which has the hydrogen atom combined with the silicon atom concerned is said, in this detail in the letter, it is alkane and sets, and for convenience, when two hydrogen atoms (H) have combined with the same silicon atom (Si), it calculates with two hydrosilyl radicals.

[0021] (B) A polyorgano hydrogen siloxane is mentioned as a desirable example of the compound of a component. A polyorgano hydrogen siloxane means the siloxane compound which has a hydrocarbon group or a hydrogen atom on a silicon atom. A siloxane compound may be the shape for example, of a chain, or annular, and is shown by the following concrete for example, formulas.

[0022]

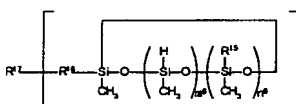
[Formula 1]

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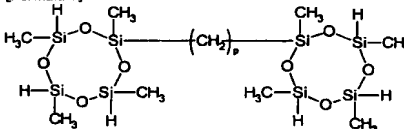
(It is  $1 \leq m \leq 6$ , is  $0 \leq n \leq 6$ , and is  $3 \leq m+n \leq 50$ , and R15 is the hydrocarbon of carbon numbers 2-20, and R15 may be permuted by one or more phenyl groups if needed.) It is  $2 \leq k \leq 3$ , and R16 is a divalent organic radical, or R16 does not need to exist. R17 is the organic radical of 2 - tetravalence.

(B) As for a component, what has good compatibility with the (A) component, the (C) component, and the (D) component or the distributed stability in a system is desirable. If compatibility with each above-mentioned component of the (B) component is low when especially the viscosity of the whole system is low, phase separation will tend to happen and it will be easy to cause poor hardening. For this reason, in order to prevent phase separation, a filler with small particle size, such as an impalpable powder silica, may be blended as a distributed assistant.

[0028] (A) The following concrete for example, compounds are mentioned as a (B) component with comparative good compatibility or distributed stability with a component, the (C) component, or the (D) component.

[0029]

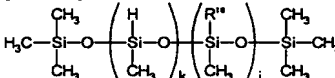
[Formula 7]



(Here, p is the integer of 6-12.)

[0030]

[Formula 8]



(It is  $2 \leq k \leq 10$ , and is  $0 \leq j \leq 5$ , and R18 is a with a carbon numbers of eight or more hydrocarbon group.)

(B) As for the amount of the component used, it is desirable to use it to the total number of mols of the alkenyl radical of the (A) component, so that the total number of mols of the silicon atomic union hydrogen atom of the (B) component may become 0.8-5.0Eq. To the alkenyl radical total amount of the above-mentioned (A) component, when there are too few silicon atomic union hydrogen atoms of the (B) component, bridge formation tends to become inadequate. Moreover, in many [ too ], a silicon atomic union hydrogen atom tends to remain after hardening, and physical properties tend to change with the effects of the hydrogen atom a lot. It is desirable to make the amount of the (B) component into 1.0-2.0Eq to control especially this effect.

[0031] Here, the substituent of the above-mentioned general formula is explained.

[0032] A hydrocarbon expresses an alkyl group, an alkenyl radical, an alkynyl group, an aryl group, an aralkyl radical, and a cycloalkyl radical preferably, when there is no notice especially.

[0033] as an alkyl group — desirable — carbon numbers 1-30 — more — desirable — carbon numbers 1-20 — the methyl of carbon numbers 1-10, ethyl, propyl, etc. are mentioned still more preferably.

[0034] as an alkenyl radical — desirable — carbon numbers 2-30 — more — desirable — carbon numbers 2-21 — the vinyl of carbon numbers 2-10, an allyl compound, etc. are mentioned still more preferably.

[0035] as an alkynyl group — desirable — carbon numbers 2-30 — more — desirable — carbon numbers 2-20 — the ethynyl of carbon numbers 2-10, propargyl, etc. are mentioned still more preferably.

[0036] as an aryl group — desirable — carbon numbers 6-30 — more — desirable — carbon numbers 6-20 — the phenyl of carbon numbers 6-10, tolyl, naphthyl, etc. are mentioned still more preferably.

[0037] as an aralkyl radical — desirable — carbon numbers 7-30 — more — desirable — carbon numbers 7-21

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- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

## EFFECT OF THE INVENTION

[Effect of the Invention] It became possible to be able to lower the degree of hardness of the elastic layer of a conductive roller, and to improve brittleness by this invention, by using into a molecule two or more sorts of compounds which have two or more hydrosilyl radicals.

[Translation done.]

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